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Shell Rolling Stability Apparatus and Shell Micro Penetrometer

R. P. McFarlane

Senior Lubricating Oil Chemist, Shell Oil Company, Incorporated,
Wood River Refinery, Wood River, Illinois.

In order to aid the development of improved ball and roller bearing greases, the "Shell Rolling Apparatus" has been designed to permit a measurement in the laboratory of the tendency of a lubricating grease to liquefy while in service. In principle the apparatus consists of a weighted iron roller enclosed in an iron cylinder which rotates at a speed of 160 r.p.m. The test is continued for a period of four hours, during which time the iron roller revolves along the wall of the cylinder intensely kneading the sample of grease.

In Figure 1 is shown a photograph of the Shell Rolling Apparatus completely assembled. Figure 2 gives details of the construction of the individual parts. Originally, it was standard practice to place about 70-75 grams of grease in the cylinder, insert the roller and run for four hours at 160 r.p.m. At the end of the test the sample was removed and the amount of breakdown or liquefaction of the grease judged by visual observation. While this examination proved satisfactory for extreme cases of good and bad stability, samples showing intermediate degrees of liquefaction remained a source of disagreement. Therefore, it became apparent that a satisfactory numerical evaluation be devised which would permit duplication and reproduction by different operators and by different laboratories. Since the accepted method for determining the consistency of a lubricating grease has been the Standard ASTM Worked Consistency, it was believed that a micro method which would show good correlation with the Standard ASTM Penetrometer should be used. As all attempts to find a micro method already in use failed, it became necessary to devise a miniature penetrometer which could be used in conjunction with the Standard ASTM Penetrometer.

An aluminum cone and cup were designed somewhat similar in shape to the ASTM cone and cup but reduced in size and weight in order to maintain the same ratio between cone diameter and the surface area of the grease in the cup and the depth of cup as now exists between the cone and cup of the Standard ASTM instrument. Because of machining difficulties no attempt was made to duplicate the double cone effect of the ASTM cone but a true cone of aluminum was fashioned (Fig. 3). Grease cups were made from four ounce seamless ointment cans used in the laboratory for asphalt penetration cups All that was necessary was to trim the top down to obtain the proper depth (Fig. 4).

As noted in table below, the theoretical value for the weight of the micro cone plus the assembly should be 30.2 grams. However, this reduction could not be obtained using the standard penetrometer. The

(Continued on page 4)

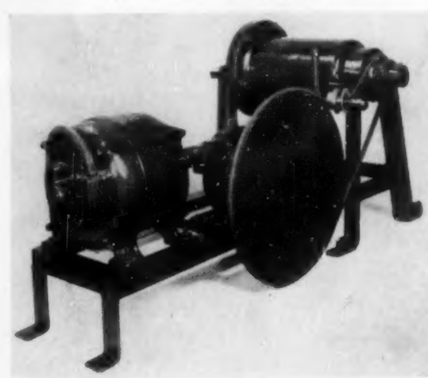

Figure 1

Data on the relative size of the Shell Micro penetration cone and cup are tabulated below in comparison with data on the Standard ASTM cone and cup.

	ASTM		*Shell Micro*	
	Cone	Cup	Cone	Cup
Volume, cc	7070	4778	1442	2551
Volume, cc	....	190	....	74
Diameter, mm	65	78	34	56
Height, mm	45	....	21	....
Depth, mm	....	65	....	30
Weight, grams (plus assembly)	150	....	58.3*	....
Surface, sq. mm	7070	4778	1442	2551
Weight in grams of cone plus assembly per sq. mm surface	0.021	....	0.040	....

*Theoretical value should be 30.2 grams.

Slushing Type Rust Preventives

By S. L. BISHKIN, Humble Oil and Refining Company, Houston, Texas

(Continued from February issue)

It is believed that the main component of lanolin is a mixture of esters of cholesterin ($C_{27}H_{45}OH$), an alcohol, and fatty acids. Apparently these esters consisting of 50 or more carbon atoms provide a surface film of high protective quality. The highly polar molecules of this type are absorbed on surfaces and adjust themselves in a closely packed fashion. (92)*.

Hundreds of other references on the subject of rust preventives were reviewed but most of them had to do with hard drying coatings. Only patent references were considered in the literature of countries other than England and the United States.

(b) Books: The following books contain information on this subject worthy of consideration:

- Protective Coatings for Metals—Burns and Schuh (E)*
  (A.C.S. Monograph No. 79—has a good review of slushing coatings).
- Corrosion of Iron and Steel—Hudson (English) (F)*.
- Pipe Corrosion and Coatings—Larsen (G)*.
- Bibliography of Corrosion — Nathan Von Patten (Canadian) (H)*.
- Bibliography on Corrosion Protective Coatings—American Documentation Institute No. 1436 (I)*.
- Tests on Protective and Decorative Coatings — Amer. Soc. Test. Mat. (J)*.
  (Symposium on Correlation between accelerated laboratory tests and service).

2. *Patents:* A rough survey of the patent literature was made by means of the chemical abstracts. Only the period 1917 through 1941 was considered. The compositions of the coatings studied were arranged first according to the main ingredient and then subdivided alphabetically by the other principal ingredients. For example, in Figure 18 all of the petrolatum coatings are grouped together. To determine what other ingredients are in a certain composition, reference should be made to the extreme left column in which these ingredients are listed alphabetically. A specific illustration is "Petroleum Oil," the fifth item. The amount of oil in this composition is shown as 85%; in the second column, the amount of the petrolatum, in the third column, the amount of other ingredients, e.g. drying oil 10% and an unspecified amount of drier; in the fourth column the use for which this composition is recommended; and in the fifth column the reference, 55. The patent from which the composition was taken may be found by noting reference No. 55 in the Appendix.

The composition is shown in subsequent charts for coatings containing soap and sulfonic acid, Figure 19; amines, Figure 20; wool fat, Figure 21; and miscellaneous materials, Figure 22.

Comments: Properly formulated and correctly applied blends of petrolatum and mineral oils have been used extensively because of the relatively good protection given and because of the low cost and availability. The protective nature of such blends varies with the viscosity and the amount of other proven additives, such as rosin, certain waxes, and oxidized petroleum residue. Polar compounds such as fatty acids, oils, alcohols, and the like are inferior to those derived from saturated hydrocarbons or mineral oil; however, spermaceti wax and lanolin are satisfactory. Some inhibitors, such as sodium dichromate, have proved unsatisfactory in numerous instances. One reason is that the sodium dichromate is normally emulsified in oily type material, in which form it is inef-

FIGURE 19

COMPOSITIONS OF SLUSHING TYPE RUST PREVENTIVES

Coatings Containing Soap and Sulfonic Acid

Other Important Ingredient	*Name of Soap and Amount*	*Other Ingredients*	*Use*	*Reference*
Aromatic Hydrocarbon	Chromium Naphthenate		General	22
Condensation Products (99-65%) of Chlarinated Wax and Aromatic Hydrocarbon	Mahogany Soap 1-35%		General	25
Hydrocarbon Oil	Sulfonic Acid 3-35%		General	60
Lard—73.5%	Aluminum Stearate 10%	Butyl Alcohol 10% Triethanolamine 6.5%	Interior and Exterior of Airplane Engine Surfaces	32
Lubricating Oil	Manganese Oleate or Manganese Chromate		To clean and prevent rust	23
Mineral Animal or Vegetable Oil	Nickel Oleate or Chromat (Colloidal Type) 1%		Lubricant which prevents and removes rust	13
Mineral Grease (Petrolatum)	Aluminum Stearate		Temporary Protection Airplane Motors	29
Montan Wax	Sodium Sulfonate	With or without mineral oil	Ferrous Metallic Surfaces	62
Petrolatum	Aluminum Soap of Coconut Oil	Paraffin Oil Potassium Hydroxide Potassium Chromate	General	73
Spindle Oil, etc. 81 pts.	Sulfonic Acid (oil insoluble) 15%		Metallic Machinery in contact with Aqueous Medium	12
Water—47 pts. (trace of formaldehyde)	Soap—16 pts.	Sodium Carbonate 8 pts. Glycerol—10 pts.	General	68
Wool Fat	Alkali Sulfonate			91

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Slushing Type Rust Preventives

(Continued from Page 2)

fective. It has also been found by a number of investigators that mineral solvents are better than chlorinated hydrocarbons for slushing type rust preventives. This is explained by the reaction of entrained moisture with the chlorinated hydrocarbons to form corrosive materials. Amines, some of which are shown in the compositions in Figure 20,

FIGURE 20

COMPOSITIONS OF SLUSHING TYPE RUST PREVENTIVES

Coatings Containing Amines

Amines (Full Name)	*Amount*	*Other Ingredients*	*Use*	*Reference*
Amine, Thiodi-alpha naphthylamine (and similar compounds)			Special Alloys Bearings	39
Amyl Amine	5-10 pts.	Sodium Hydroxide 10-15 pts. Water — 1000 parts	Rust proofing iron	70
Benzyl Amine (or other aralkylamine)			Inhibitor Ferrous Metals Contact with Water or Alcohol	30a
Dibutylthiourea (or other similar types)		Plus Lubricating Oil	Special Alloy Bearings	35
Diamino Diphenyl Amine		Mineral Hydrocarbon Oil	Special Alloy Bearings	40
Diamino Polyaryl Methane		Lube Oil	Special Alloy Bearings	36
Thio-dinaphthyl Amine	0.2% or more	Mineral Hydrocarbon Oil	Special Alloy Bearings	43
Tri-ethanol Amine	6.5%	Lard 73.5% Butanol 10% Aluminum Stearate 10%	Interior and Exterior Airplane Surfaces	32
Triethanolamine Oleate Triethanolamine	1.5% 0.5%	Oil 98%	Interior and Exterior Airplane Surfaces	32a

FIGURE 21

COMPOSITIONS OF SLUSHING TYPE RUST PREVENTIVES

Coatings Containing Wool Fat

Other Important Ingredients	*Other Ingredients*	*Use*	*Reference*
Carbon Tetrachloride of Trichloroethylene		For General Use	5
Oil, Lube		Lubricant and Rust Preventive	78
Paraffin up to 10%	Ceresin up to 3% if in White Spirits Solution	For General Use	B (Pg. 9)
Petrolatum	Lanolin Spindle Oil Machine Oil	Steel	15
Resin, Hard (Hydrocarbon Soluble)	May also include Petroleum Jelly, Paraffin, Beeswax, Carnauba, Pigments	For General Use	28 & 31
Solvent, Non-Acid Non-Volatile (or Mineral Hydrocarbon) or Lube Oil		For General Use	61
Sulfonate, Alkali		For General Use	91
Spirits, White (or Solvent Naphtha)—50%		For General Use	B (Pg. 18)

(Continued on Page 6)

(Continued from Page 1)

higher value of 58.3 grams can easily be obtained by adjusting the lead shot in the penetrometer shaft. From the data collected it appeared desirable to use the higher value since it requires a heavier weight to drive the micro cone to a depth proportional to the depth reached by the double cone of the ASTM apparatus.

In Figure 5 is shown a photograph of the Shell Micro Penetrometer Assembly which illustrates the simplicity of the conversion of the Standard ASTM instrument. Figures 3 and 4 give in detail the dimensions of the Aluminum cone and tin cup. Tests are run using the same method specified by the ASTM for Consistency of Lubricating Greases and Petrolatums ASTM Designation D-217-38T.

To show the correlation between the Shell Micro Penetrometer and the Standard ASTM Penetrometer a number of tests were made using both instruments. Greases tested comprised all the various types found in general use. The results of these tests are tabulated in the following table:

The curve drawn from the above data (Figure 6) shows that the relationship between the Shell Micro and the Standard ASTM Penetrometers is quite satisfactory and that it is not adversely affected by the different types of soaps present.

In view of the above correlation, it is believed that the Shell Micro Penetrometer

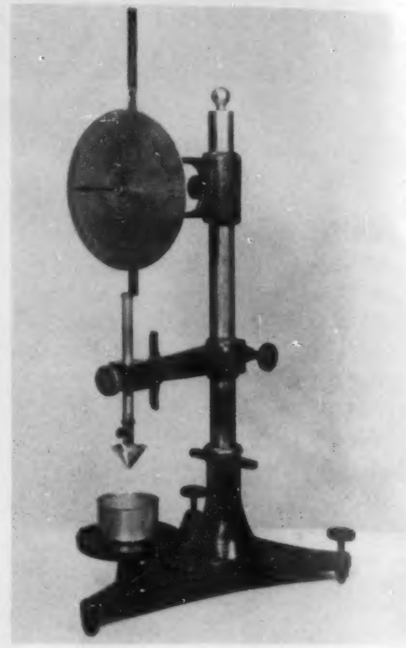

FIGURE 5

is a valuable adjunct to the ASTM instrument for determining consistencies of quantities too small to be obtained by the regular ASTM method.

As a result of the development of a satisfactory micro penetrometer the evaluation of greases, subjected to the Shell Rolling Stability Test, is now simplified. The micro penetrometer permits a definite expression of values in units which can be converted to ASTM Penetration Units [*] by means of the curve designated Figure 6. The procedure for running the Shell Roll-

(*) ASTM D-217-38T expresses depth of penetration in tenths of a millimeter.

	Worked Consistency of Grease	
Grease	*ASTM*	*Shell Micro*
N. L. G. I. No. 1 Calcium Grease	335	180
N. L. G. I. No. 3 Sodium Grease	240	100
N. L. G. I. No. 1 Calcium Grease	325	175
N. L. G. I. No. 3 Calcium Grease	230	90
N. L. G. I. No. 4 Calcium Grease	187	58
N. L. G. I. No. 1 Aluminum Grease	300	144
N. L. G. I. No. 1 Calcium Grease	325	167
N. L. G. I. No. 2 Calcium Grease	269	118
N. L. G. I. No. 2 Calcium Grease	270	123
N. L. G. I. No. 2 Calcium Grease	275	121
N. L. G. I. No. 2 Calcium Grease	270	120
N. L. G. I. No. 2 Calcium Grease	267	115
N. L. G. I. No. 2 Calcium Grease	265	114
N. L. G. I. No. 4 Calcium Grease	205	70
N. L. G. I. No. 5 Calcium Grease	139	37
N. L. G. I. No. 3 Aluminum Grease	243	105
N. L. G. I. No. 3 Aluminum Grease	250	107
N. L. G. I. No. 4 Aluminum Grease	205	70
N. L. G. I. No. 1 Sodium Grease	340	185
N. L. G. I. No. 1 Sodium Grease	332	188
N. L. G. I. No. 1 Calcium Grease	336	182
N. L. G. I. No. 1 Sodium Grease	337	191

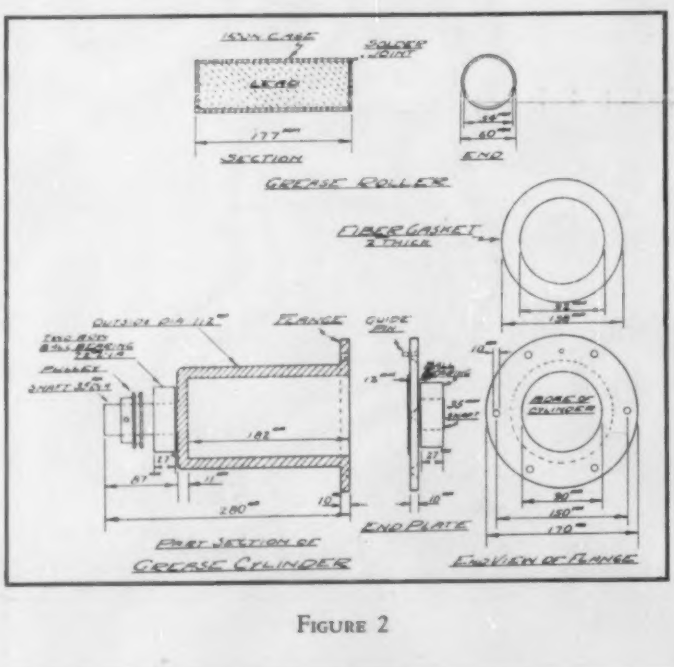

FIGURE 2

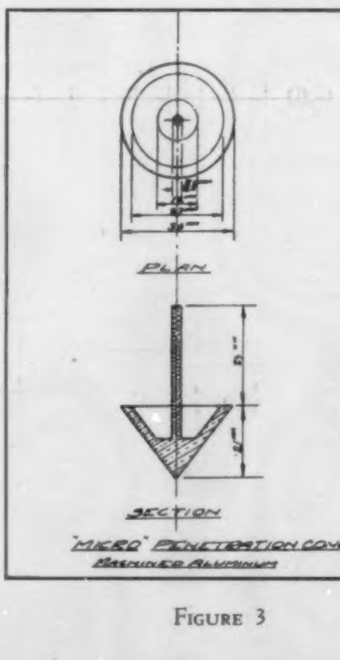

FIGURE 3

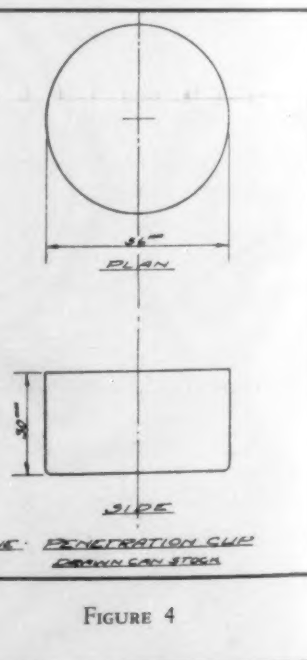

FIGURE 4

ing test is as follows: After the grease sample is rolled four hours, the rolled grease is transferred to the Micro Penetration cup and allowed to stand in a 77°F. bath for not less than 10 nor more than 15 minutes. At the end of that time the Micro penetration is determined. Greases showing more than 80 points difference between unrolled and rolled portions are considered as having undergone abnormal softening and should be classified as having unsatisfactory roller stability.

To check the reproducibility of both the Shell Rolling Stability Test and the Shell Micro Penetration Test, samples of greases were submitted to two laboratories. The results of their tests are tabulated below:

N. L. G. I. No.	*1*		*3*		*3*	
Type of Soap	*Calcium*		*Sodium*		*Sodium*	
Laboratory No.	*1*	*2*	*1*	*2*	*1*	*2*
A S T M Worked Cons. 77° F.	338	340	248	250	232	227
Duplicate	337	338	253	250	230	227
Average	338	339	251	250	231	227
Shell Micro Cons. 77° F.	168	172	102	102	84	92
Duplicate	164	167	105	103	88	93
Average	166	169	103	103	86	93
Shell Rolling Stab. Test						
Micro Cons. 77° F.	170	177	158	141	140	128
Duplicate	180	172	156	156	150	156
Triplicate	.....	181	.....	158	.....	146
Average	175	177	157	152	145	143

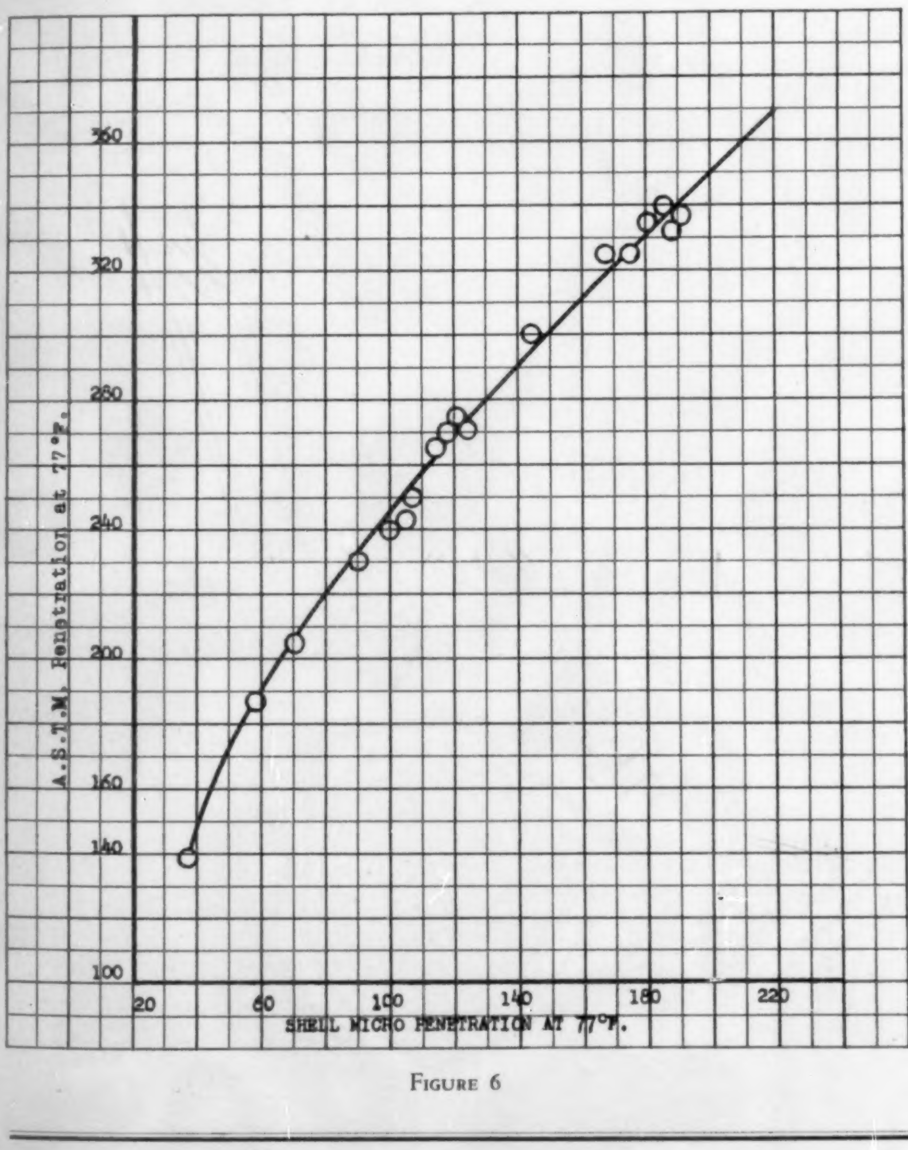

Figure 6

Conclusion

While the Shell Rolling Apparatus will not evaluate all the properties of greases which relate to service performance, it is felt that more than 80 units breakdown, as measured by the Shell Micro Penetration Method, indicates that undue breakdown or liquefaction due to mechanical working in service may be expected, which will result in excessive leakage. The availability of the apparatus used in the Shell Micro Penetration Method and the excellent correlation with the Standard ASTM Instrument are noteworthy. Not only is the Shell method valuable in evaluating greases after being subjected to the Shell Rolling Test but it has been of value in obtaining information on small samples of lubricating grease removed from anti-friction bearings of motors where only relatively small quantities were used and where the available samples were inadequate for measurements by the usual ASTM Method.

Comments

Pertinent comments have been made by several users of this equipment which covers experience in these tests.

"When using the machine we occasionally had difficulty with the inside roll, which seemed to form a suction in the grease and did not roll properly. We overcame this difficulty by placing a smaller plug inside the large cap. This plug can be removed, thus preventing any pressures or vacuum to be formed in the larger tube. The inside roll can be tapped occasionally to prevent sticking."

"The Roll Test is an excellent test, and we feel that it has given us a splendid means by which we can predict the actual performance in service of our greases."

Further comment is with reference to the taking of the penetration of the rolled grease. "This should be brought to the required 77°F. temperature and the penetration taken within a period of at least ten minutes after the completion of the roll test to overcome any tendency of the "setting up action" of the grease after rolling."

Further work has been done with reference to running tests at various temperatures, and no doubt this work will be reported at a later date. . . .

FIGURE 22 — COMPOSITIONS OF SLUSHING TYPE RUST PREVENTIVES — *Other Miscellaneous Coatings*

Principal Ingredient	*Amount*	*Other Ingredients*	*Use*	*Reference*
Acid, Oleic (or higher Carboxylic Acid)		Lubricating Oil	For Internal Combustion Engines	16
Acid, Oleic (Stearic or Palmitic)		Water, Glycerol and Ethylene Glycol	For Iron	58
Acid, Phosphoric, Diphenyl		Refined Mineral Oil	For Steel Sheets	2
Alcohol, Butyl (Secondary)		Potassium Hydroxide (with or without EtOH); Fatty Acids; Petroleum Solvent; Pigments	For Iron and Steel	20
Alizarin (or similar material)		plus Lubricating Oil	For Bearings	37
Amine, Benzyl (or Aralkyl Amine)			Inhibitor Ferrous Metals in Contact with Water or Alcohol	30a
Arsenite, Tributyl or Triamyl	0.2% or less	Lubricating Oil	For Special Alloy Bearings	34a
Benzonitrile	0.2% Minimum	Lubricating Oil	For Special Alloy Bearings	45
Carbonate, Sodium	88%	Ammonium Sulfate 12%; and enough Oil to form Paste	For Battery Terminals	56
Chloride, Zinc	48 Parts	Glycerol 4 Parts; Zinc Sulfate 2 Parts; Water 46 Parts	For Wire Wheels, Auto Rims	74
Cresol		Lubricating Oil	For Internal Combustion Engines	48
Grease, Axle	70-80 Parts	Rape Oil 10-20 Parts; Concentrated Water Glass Solution 5-10 Parts	For General Use	82
Lard	73.5%	Butanol 10%; Aluminum Stearate 10%; Triethanol Amine 6.5%	Interior and Exterior Airplane Surfaces	32
Lard	16 Parts	Black Lead 4 Parts; Camphor 1 Part	For Bright Metals	69
Morpholine (derivative)	0.5-0.2%	Plus Mineral Oil	For Special Alloys	46
Oil		Graphite Thinner (or heavy Grease) Odorant	For Heavy Bearings	90
Oil, Castor		Alcohol, with or without Phosphoric Acid and Fusel Oil		7
Oil, Cylinder	80%	Ceresin 20%		21
Oil, Drying (gelatinized)		Semi-Drying Oil; Mineral Oil; Drier	For General Use	27
Oil, Lard	25 Parts	Lubricating Oil 25 Parts; White Lead 25 Parts; Graphite 15 Parts; Borax 5 Parts; Lampblack 5 Parts	For Coating Steam Boiler or Pipes	72
Oil, Lard		Carbolic Acid	Firearms	80
Oil, Linseed (Boiled)	10%	Petroleum Oil Distillate; 2% Pine Oil; 2% Oleic Acid; Drier	Prevention and Removal	3
Oil, Lubricating	99-99.5%	Polycarboxylic Acid	Internal Combustion Engines	26
Oil, Paraffin	60-200 Parts	Fatty Acids 2 Parts; Litharge 1 Part	General	83
Oil, Paraffin Base		Camphor Dissolved in Alcohol and Alum in Water	General	89
Oil, Seal	18 Parts	Graphite 33 Parts; Horsefat 7.5 Parts Zinc White 7.5 Parts; Grease (Solid Tallow with Ca or Al Soap)	Lubricant and Rust Preventive	53
Ozokerite		Stand Oil; Pigments	For Structural Steel	81
Peroxide, Sodium (reaction with wood alcohol)		Plus Lubricating Oil	Corrosion Preventing Top Lube Oil	49 and 50
Peroxide, Tetralin	0.1% or more	Petroleum Oil	For Copper	4
Phosphate, Calcium (precipitated)		Oil		66
Phosphites, Alkyl		and Lubricating Oil	For Special Alloy Bearings	17
Phosphites, Aryl		Lubricating Oil	For Special Alloy Bearings	18
Pitch (from Oils and Fats)	30-40 Parts	60-70 Parts Tar Distillate or Hydrocarbon Solvents (See also U.S. 1,379,019 which includes TiO_2		67
Sulphur	0.1-0.5%	Plus Lubricating Oil	For Special Bearings	42
Tar Oil	68 Parts	Asphaltum 20 Parts; Stearin Pitch 12 Parts	For Iron or Steel	8
Thiourea (condensation of Thiourea and Mustard Oil) etc.		Lubricating Oil		41
Urethan-cyclic				1
Zinc		Semi-Solid Mixtures of Paraffin or Olefin Grease	For Steel Ropes in Acid Water	51

Slushing Type Rust Preventives

(Continued from Page 6)

are helpful as neutralizing agents for acidic products. Zinc dust has been found effective in many practical applications as a component of neutral petrolatum blends.

Not much literature was available on the efficiency of most of the ingredients included in Figures 18 through 22. A few comments are made above about some of the ingredients of the slushing compounds. It is safe to say that many of the materials used in the slushing compositions are worthless, and a number which are effective have limited application. The information supplied on these compositions should find utility as a starting point in an investigation of slushing compounds as it provides a cross section of the patents in this field.

3. *Summary of Principal Government Specifications on Slushing Compounds:* Considerable information on slushing compounds is found in Government specifications. For quick reference, the principal slushing compounds used by the various Government agencies are analyzed in Figure 23. The branches of the Government considered are: Army, Navy, Maritime Commission, Air Corps, and Ordnance. For example under Navy Specification OS-627 is shown the date on which the specification was made effective, July 30, 1940; the grade and the consistency, Grade A solid; use, for guns; length of service expected, one year; and whether the approval of the compound is based on laboratory or service tests, in this case, both. In similar style the other principal Government specifications are reviewed. A number of different types are shown, such as heavy pigmented grade in Navy 14-C-6A to the light polar unpigmented liquid as covered by Navy Bureau of Ships 52-C-18(INT).

Revised specifications have likely been issued since the table was prepared. The writer received Army-Navy Specification ANC 52 of August 31, containing a compound similar to 2-84-b and also a leaded composition neither one of which is included in this review.

The length of service expected varies from ten years in specification Army 2-82C to two months for specification 14-C-4d compound, rust preventive, Type B, quick drying. This latter composition is in reality not a slushing compound, in the usual sense of the word. A service test is required for the Government approval on a limited number of rust preventives, for example Navy OS-627. In some instances, however, the Government will waive the service test if the vendor supplies an affidavit that the slushing compound has given comparable service in other applications.

(To be continued)